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13. ABSTRACT (Maximum 200 words)

The research proposed here is based on the principle of the density gradient cloumn. 1 A liquid (A) of low density is continuously mixed into a liquid (B) of higher density while B is allowed to flow slowly down the wall of a glass cylinder. The feed rate of A is equal to the flow rate of mixture. Thus, a gradient density column is formed. Such columns have been used to measure the density of semiconductors to five (5) significant figures. The gradient is stable is stable for many months at room temperature. We proposed to use this method to prepare gradient index (GRIN) lenses from gels with large axial gradients. The chemical compositions of two sols are selected based on considerations of solubility between the sols; differences in refractive index, density, expansion coefficient and densification temperatures between resulting oxides.

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Air Force Office of Scientific Research

for project entitled

GRADIENT INDEX LENSES FROM SOL-GEL LAYERING
(AN AASERT AWARD)

Grant No.: F49620-93-1-0364
Inclusive Dates: 1 July 1993 to 30 June 1994

PRINCIPAL INVESTIGATOR

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1. Introduction and Background

This is an AASERT grant award with an official starting date of July 1, 1993. One female graduate student, Miss Tammy Chau, who is a U.S. citizen is being supported by this grant.

The research proposed here is based on the principle of the density gradient column.[1] A liquid (A) of low density is continuously mixed into a liquid (B) of higher density while B is allowed to flow slowly down the wall of a glass cylinder. The feed rate of A is equal to the flow rate of the mixture (Figure 1). Thus, a gradient density column is formed. Such columns have been used to measure the density of semiconductors to five (5) significant figures. The gradient is stable for many months at room temperature. We proposed to use this method to prepare gradient index (GRIN) lenses from gels with large axial gradients. The chemical compositions of two sols are selected based on considerations of solubility between the sols; differences in refractive index, density, expansion coefficient and densification temperatures between resulting oxides.

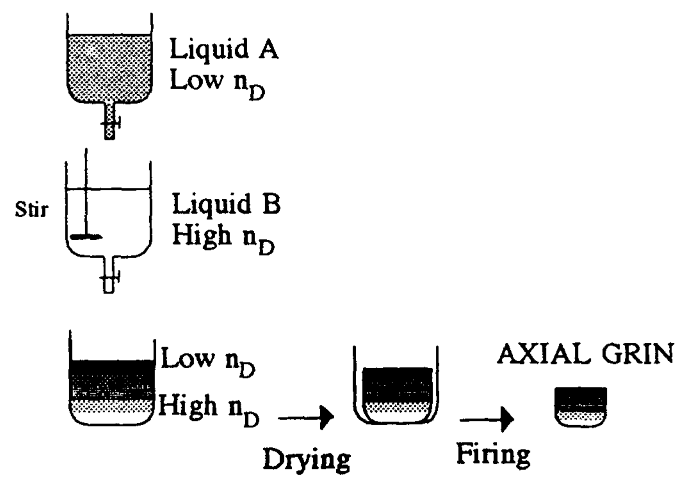


Figure 1-The density column approach for the fabrication of gel-derived axial GRIN

2. Research Performed

A- The TiO_2-SiO_2 system.

Initial experiments were carried out on the TiO_2-SiO_2 binary system. Pure SiO_2 has a refractive index of 1.46, and a 15%TiO_2-85%SiO_2 composition would have a theoretical refractive index of 1.56, giving a Δn of 0.1 from end to end of a rod. Two solutions, one of pure tetraethyl orthosilicate (TEOS) and one containng titanium isopropoxide (0.9 TEOS - 0.1 titanium isopropoxide), were prepared (Figure 2). The solutions were gradient-cast according to the process described in Figure 1. After careful drying and firing (up to 900°C) at a heating rate of 0.5°C/min, the rods were sliced perpendicular to the optical axis for refractive index measurements. The oil immersion technique was used. Figure 3 shows the gradient in refractive index of a 5.62 mole% TiO_2 fired gel. Gels containing up to 15% TiO_2 have been prepared with this process. However, they did not retain their transparency as TiO_2 crystals formed during heat treatment. It was therefore decided that an alternate system, which could give a large Δn without risk of precipitation of a second phase, be investigated.

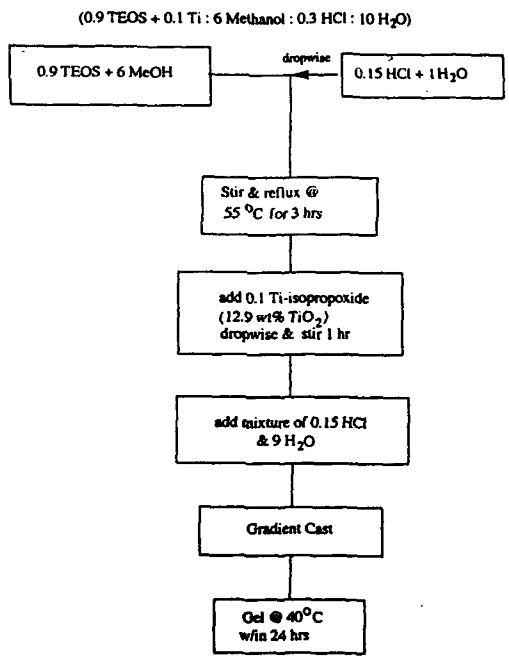


Figure 2- Flow-chart for the preparation of TiO_2-SiO_2 gels

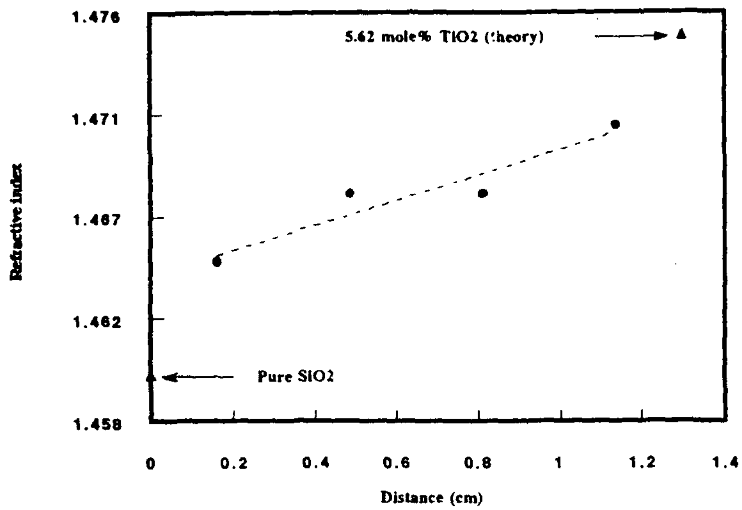


Figure 3 - Gradient in TiO_2-SiO_2 gel-derived axial GRIN rod

B- The TiO_2-PbO system

TiO_2-PbO monoliths have recently been fabricated by the sol-gel technique[2]. Although these materials were initially investigated for their high non-linear optical coefficients, they are of great relevance to our study because they also exhibit a large variation in refractive index with composition. Transparent glasses have been fabricated over a wide compositional range (Figure 4). The refractive index of a 60%mole TiO_2-40% mole PbO is 2.030, and that of a 80% TiO_2-20% PbO is 2.268.

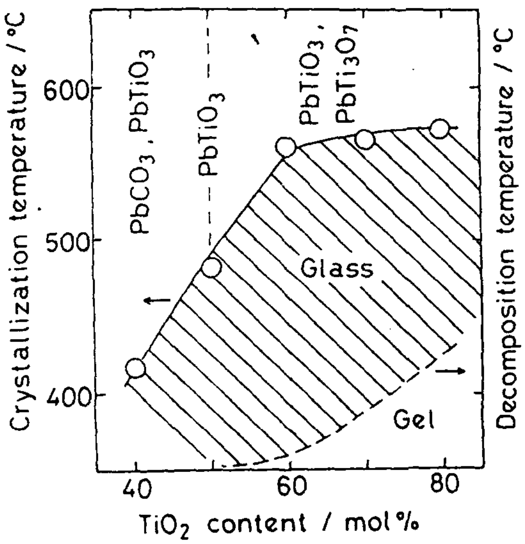
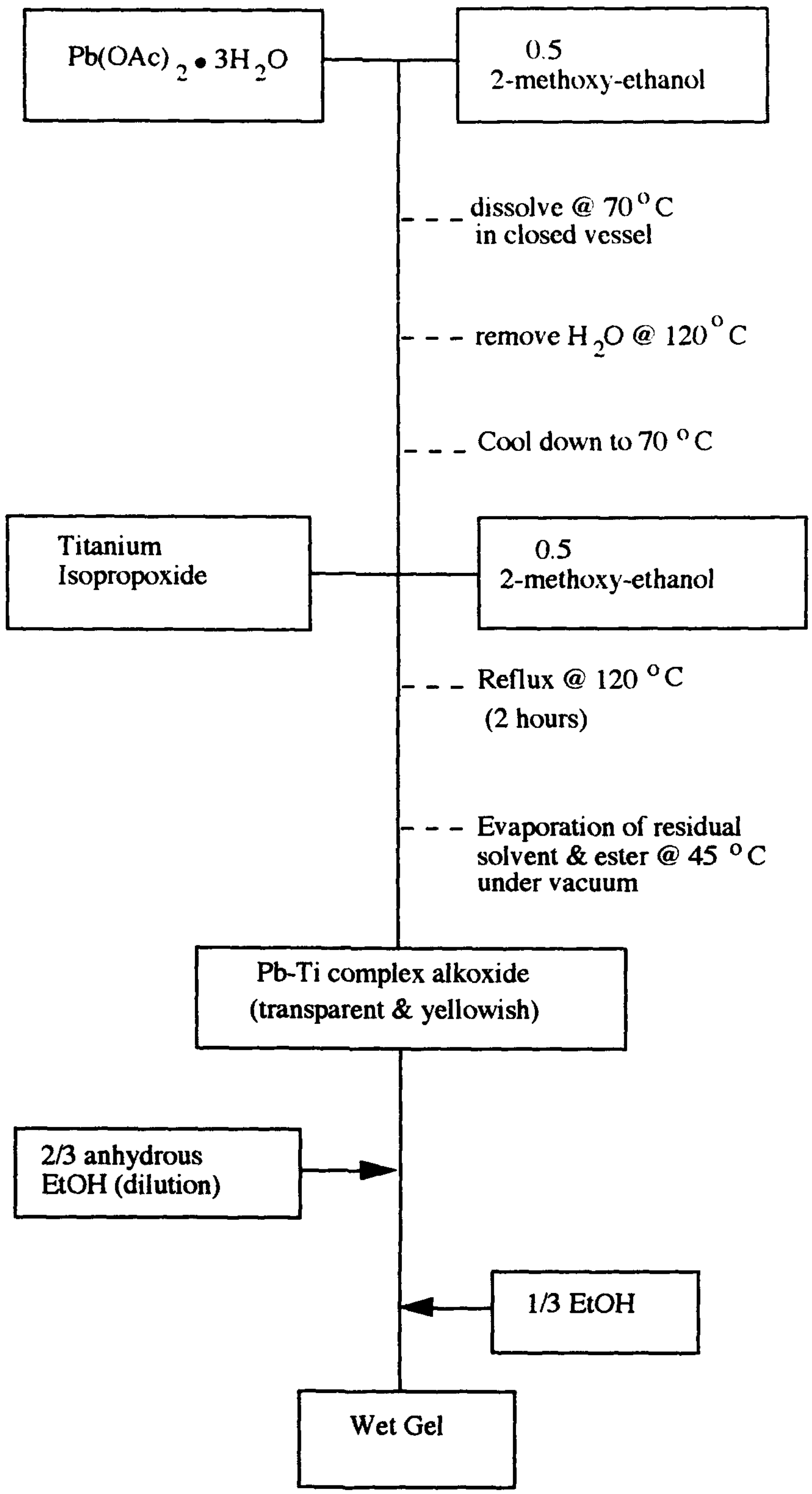


Figure 4 - Glass forming region of TiO_2-PbO gels [2]

Figure 5- Flow chart for the synthesis of $PbO\text{-}TiO_2$

(1 mole alkoxide : 4 moles 2-methoxy ethanol : 6 moles ethanol)



Two solution compositions were prepared, one containing 80% TiO_2 (solution A), the other 60% TiO_2 (solution B), were prepared according to the flow-chart in Figure 5. Using the set-up previously described, a gradient gel was cast in polypropylene containers. The refractive indices of gels of both extreme compositions are presented in Table 1. These values were obtained by ellipsometry on thin films (0.2-0.4 μm) deposited on silicon substrates by spin-coating. The maximum Δn measured was 0.38 for a gel dried at room temperature and 0.28 for a gel fired at 500°C for 1/2 hour.

Table 1- Refractive indices of PbO-TiO_2 gels

	80TiO_2-20PbO	**60TiO_2-40PbO**	**Δn**
n_D (gel)	**1.77**	**2.15**	**0.38**
n_D (500°C)	**2.84**	**2.56**	**0.28**

The Δn value of the fired gels are in agreement with that reported by Nasu et al. The existence of an axial gradient has been demonstrated by observing the path of a laser beam through a wet gel. As one can see from Figure 6, the laser beam is bent in passing through the gradient gel.

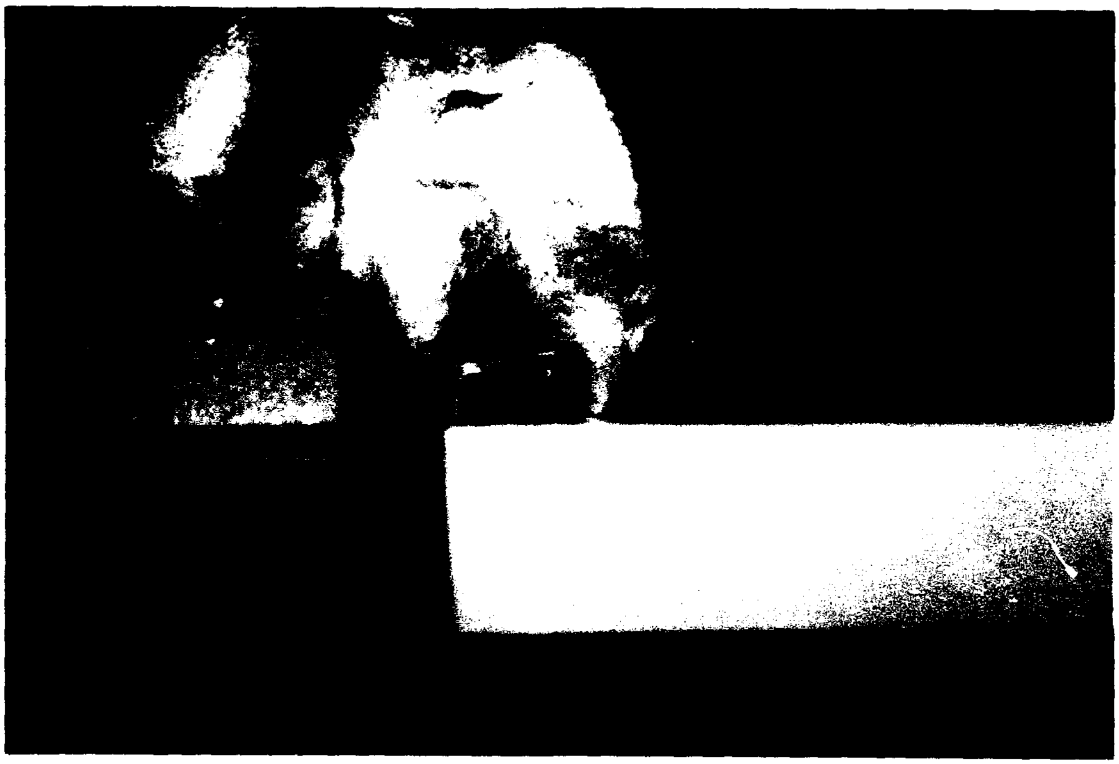


Figure 6 - Photograph of light bending in a PbO-TiO_2 GRIN gel

FUTURE WORK

The feasibility of an axial gradient index material by the sol-gel technique has been demonstrated for the first time. Future work will be aimed at drying and firing gel to obtain a GRIN glass. Some important parameters involved in fabrication such as solution concentration and viscosity, will be investigated. The gradient index in the glass will be optimized through appropriate modification of the starting solution. Based on the promising results of the $PbO\text{-}TiO_2$ system, we also intend to investigate the $PbO\text{-}SiO_2$ system which would exhibit many advantages over the $PbO\text{-}TiO_2$ system.

REFERENCES

1. F. H. Horn, Phys. Rev. 97, 1521 (1955)
2. H. Nasu, K. Kamiya, Y. Katagiri, S. Makino and J. Matsuoka, Sol-gel Optics III, SPIE Proc. Vol 2288 (1994), in print